Ann. Occup. Hyg., Vol. 54, No. 6, pp. 697–709, 2010

doi:10.1093/annhyg/meq050

Performance of High Flow Rate Samplers for Respirable Particle Collection

TAEKHEE LEE[1]*, SEUNG WON KIM[1], WILLIAM P. CHISHOLM[1], JAMES SLAVEN[2] and MARTIN HARPER[1]

[1]Exposure Assessment Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention, Morgantown, WV 26505, USA; [2]Biostatistics and Epidemiology Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention, Morgantown, WV 26505, USA

Received 9 March 2010; in final form 29 April 2010; published online 21 July 2010

The American Conference of Governmental Industrial hygienists (ACGIH) lowered the threshold limit value (TLV) for respirable crystalline silica (RCS) exposure from 0.05 to 0.025 mg m^{-3} in 2006. For a working environment with an airborne dust concentration near this lowered TLV, the sample collected with current standard respirable aerosol samplers might not provide enough RCS for quantitative analysis. Adopting high flow rate sampling devices for respirable dust containing silica may provide a sufficient amount of RCS to be above the limit of quantification even for samples collected for less than full shift. The performances of three high flow rate respirable samplers (CIP10-R, GK2.69, and FSP10) have been evaluated in this study. Eleven different sizes of monodisperse aerosols of ammonium fluorescein were generated with a vibrating orifice aerosol generator in a calm air chamber in order to determine the sampling efficiency of each sampler. Aluminum oxide particles generated by a fluidized bed aerosol generator were used to test (i) the uniformity of a modified calm air chamber, (ii) the effect of loading on the sampling efficiency, and (iii) the performance of dust collection compared to lower flow rate cyclones in common use in the USA (10-mm nylon and Higgins–Dewell cyclones). The coefficient of variation for eight simultaneous samples in the modified calm air chamber ranged from 1.9 to 6.1% for triplicate measures of three different aerosols. The 50% cutoff size ($_{50}d_{ae}$) of the high flow rate samplers operated at the flow rates recommended by manufacturers were determined as 4.7, 4.1, and 4.8 μm for CIP10-R, GK2.69, and FSP10, respectively. The mass concentration ratio of the high flow rate samplers to the low flow rate cyclones decreased with decreasing mass median aerodynamic diameter (MMAD) and high flow rate samplers collected more dust than low flow rate samplers by a range of 2–11 times based on gravimetric analysis. Dust loading inside the high flow rate samplers does not appear to affect the particle separation in either FSP10 or GK2.69. The high flow rate samplers overestimated compared to the International Standards Organization/ Comité Européen de Normalisation/ACGIH respirable convention [up to 40% at large MMAD (27.5 μm)] and could provide overestimated exposure data with the current flow rates. However, both cyclones appeared to be able to provide relatively unbiased assessments of RCS when their flow rates were adjusted.

Keywords: calm air chamber; CIP10-R; FSP10; GK2.69; high flow rate sampler; respirable fraction; sampling efficiency

INTRODUCTION

Respirable crystalline silica (RCS) exposure has been estimated to affect 1.7 million workers in

*Author to whom correspondence should be addressed.
Tel: +1-304-285-5783; fax: +1-304-285-6041;
e-mail: fwc8@cdc.gov

various industries in the USA [Occupational Safety and Health Administration (OSHA) 2004]. Exposure to RCS is associated with an increased risk of developing silicosis, tuberculosis, other nonmalignant respiratory diseases, and autoimmune respiratory diseases (Steenland and Sanderson, 2001; Ulm *et al.*, 2004; Nij and Heederik, 2005). Crystalline silica is classified as a 'Group One' human carcinogen by the International Agency for Research on Cancer (IARC, 1997). A National Institute for Occupational Safety and Health (NIOSH) hazard review report concluded that the estimated risk of silicosis for a 45-year working lifetime was 47–95% for cumulative RCS exposures at the current permissible exposure limit (PEL = 0.1 mg m^{-3}) regulated by the OSHA in the USA, and OSHA has targeted RCS under its current rule-making efforts. In addition, the NIOSH hazard review report also found a lifetime risk of silicosis approximately 10–30% when RCS concentrations are equal to the NIOSH recommended exposure limit (0.05 mg m^{-3}) (NIOSH, 2002). The Health and Safety Executive (HSE, 2002) calculated a risk of 2.5% (5 in 200 workers) of developing silicosis after 15 years of exposure to RCS at 0.1 mg m^{-3}, with a 0.5% (1 in 200 workers) risk even at 0.04 mg m^{-3}. In light of this new information on the hazardous nature of RCS, a threshold limit value (TLV) for silica of 0.025 mg m^{-3} was accepted in 2006 by the American Conference of Governmental Industrial Hygienists (ACGIH, 2006).

Two current standard sampling and indirect analysis methods for analyzing crystalline silica are NIOSH 7603 (via infrared absorption spectrometry) and NIOSH 7500 (via X-ray powder diffraction). Both methods use the 10-mm nylon cyclone (flow rate at 1.7 l min^{-1}) or the Higgins–Dewell (HD) cyclone (flow rate at 2.2 l min^{-1}) as the standard sampler for collecting respirable airborne particles for crystalline silica analysis. Both current standard sampling methods are limited to a sample air volume of $\leq$1 m^3 over an 8-h sampling time. Analytical methods for RCS have been evaluated through the Proficiency Analytical Testing (PAT) conducted by American Industrial Hygiene Association for several decades. PAT samples have been produced to bracket the silica mass anticipated from sampling 1 m^3 of an atmosphere having a concentration around the PEL value of 0.1 mg m^{-3}. The PAT results were organized and published last in 1999 for the PAT rounds conducted during the period from 1990 to 1998 (Eller *et al.*, 1999). The lower limit of loading for these PAT samples was 0.04 mg, almost twice the mass that would be collected by the same samplers over 8 h when the concentration is at the new ACGIH TLV, and, therefore, the results cannot definitively inform us of the interlaboratory precision around the new TLV. However, there is clear evidence of an increase in variance with lower loading and extrapolation of the relationship predicts an unacceptable level of variation at or below the new TLV. In addition, occupational hygienists may have to sample the environment for shorter periods than 8 h due to limitations of the working environment. A low flow rate aerosol sampling instrument ($\sim$2 l min^{-1}) that might not be able to provide sufficient samples of RCS for reliable quantitative measurements over an 8-h period certainly will not collect sufficient mass for periods $<$8 h. For the purpose of collecting more mass of respirable dust for quantitative analysis, several high flow rate aerosol samplers have become commercially available, including the CIP10-R (Courbon *et al.*, 1988; Gero and Tomb, 1988), GK2.69 cyclone (Kenny and Gussman, 1997), and FSP10 cyclone (Dahmann *et al.*, 2001). However, there are few studies focusing on the application of these high flow rate samplers to collect samples for RCS. This study aims to evaluate the performance of high flow rate samplers under controlled laboratory conditions against the international standard for respirable aerosol sampling (Soderholm, 1989) and to compare this performance to low flow rate samplers that are commonly in use in the USA as a preliminary step to determine whether these samplers are suitable for sampling respirable RCS at concentrations around the new ACGIH TLV. In order to accomplish these aims, we determined the sampling efficiencies of high flow rate samplers with monodisperse aerosols and examined the effect of increased loading on their performance. We then compared the collection of dust by high and low flow rate samplers.

MATERIALS

High flow rate samplers

The three high flow rate samplers for respirable fraction particles employed in this study were (i) CIP10-R (Arelco ARC, Paris, France), particle collection by polyurethane foam cup, sampling flow rate at 10 l min^{-1}; (ii) GK2.69 (BGI Inc., Waltham, MA, USA), particle collection by 5-μm pore size 37-mm polyvinyl chloride (PVC) filter (GLA5000; SKC Inc., Eighty Four, PA, USA), sampling flow rate at 4.2 l min^{-1}; and (iii) FSP10 (GSA (Gesellschaft für Schadstoffmessung und Auftragsanalytik) Messgerätebau GmbH, Neuss, Germany), particle collection by 5-μm pore size 37-mm PVC filter

(GLA5000; SKC Inc.), sampling flow rate at 10 l min^{-1} (Table 1).

Test particles

The mass median aerodynamic diameter (MMAD) and geometric standard deviation (GSD) of the test aerosols utilized in three different experiments are shown in Table 2. For the uniformity test of the calm air chamber, aluminum oxide grade F800 (Washington Mills, Niagara Falls, NY, USA), grade F1200 (Washington Mills), and kaolin (Fisher Scientific, Pittsburgh, PA, USA) were used. For the dust loading effect on sampling efficiency test, Air Cleaner (AC) fine test dust (Duke Scientific, Palo Alto, CA, USA) was utilized. For the mass concentration comparison between high and low flow rate samplers, aluminum oxide grades F800 and F1200 and AC fine test dust were utilized. The MMADs of aluminum oxide grade F800 and grade F1200 were determined from Aerodynamic Particle Sizer (APS; Model 3321; TSI Inc., Shoreview, MN, USA) measurements in this study. The MMADs for F1200 and AC fine test dust showed only small differences from the APS measurement. However, the AC fine test dust was found to have a bimodal distribution, i.e. it had a second peak in its size distribution between the size of 0.5 and 1.5 μm. The AC fine test dust was considered the smallest size dust in dust loading effect on sampling efficiency test.

METHODS

In this study, we used a calm air chamber whose performance has already been validated (Feather and Chen, 2003; Lee *et al.*, 2009), referred to as chamber A, as well as a modification of this chamber, referred to as chamber B, where additional space was provided for the test section. Since the uniformity of chamber B had not previously been established, experiments to evaluate it are reported here.

Calm air chamber and its uniformity test

The calm air chamber (chamber B) and experimental setup utilized in this study are shown in Fig. 1. The chamber was constructed of aluminum and measured 0.53 m diameter and 0.69 m high. The generated particles were introduced from the top with a fluidized bed aerosol generator (Model 3400; TSI Inc.) whose dry high efficiency particulate air-filtered air was provided through a filtered air supply unit (TSI model 3074). The aerosol was introduced into the chamber through a ^{85}Kr aerosol neutralizer (Model 3012A; TSI Inc.) and a special aerosol nozzle allowing the airflow to enter the chamber in a radial direction. For the uniformity test in the calm air chamber, eight 37-mm closed-face cassettes (CFCs; Omega Specialty Instruments, Houston, TX, USA) with PVC filters (5 μm pore size, GLA5000; SKC Inc.) simultaneously collected test aerosols at a flow rate of 2 l min^{-1}. Prior to the sampling, the cassettes were closed with a cassette closer (Omega Specialty Instruments, now SKC Inc.) and leakage was checked with a cassette leak tester (Omega Specialty Instruments). The filters were weighed before and after sampling in a facility with constant relative humidity and temperature. The CFCs were located symmetrically at a distance halfway between the center and the wall and faced horizontally. Dust was collected for 90 min in each measurement and tests were repeated three times for each aerosol. The flow rate for each CFC was determined as the average of the volumetric flow rate measured before and after sampling with a mass flow meter (Model 4100; TSI Inc.). Coefficient of

Table 1. Comparison of high flow rate samplers

High flow rate samplers	Flow rate (l min^{-1})	Sampling media	Sampling principle
CIP10-R	10	Polyurethane foam filter	Impaction and selective filtration
GK2.69	4.2	PVC filter (37 mm, 5 μm pore size)	Cyclone size selection
FSP10	10	PVC filter (37 mm, 5 μm pore size)	Cyclone size selection

Table 2. MMAD and GSD of test particles

	AC fine test dust	Aluminum oxide grade F800	Aluminum oxide grade F1200	Kaolin
MMAD (μm) (GSD) determined by an APS from present study	2.53 (1.95)	4.45 (1.52)	2.86 (2.01)	2.21 (1.78)
Tests used	Dust loading effect	Uniformity test for calm air chamber Sampling efficiency for GK2.69 cyclone	Uniformity test for calm air chamber	Uniformity test for calm air chamber

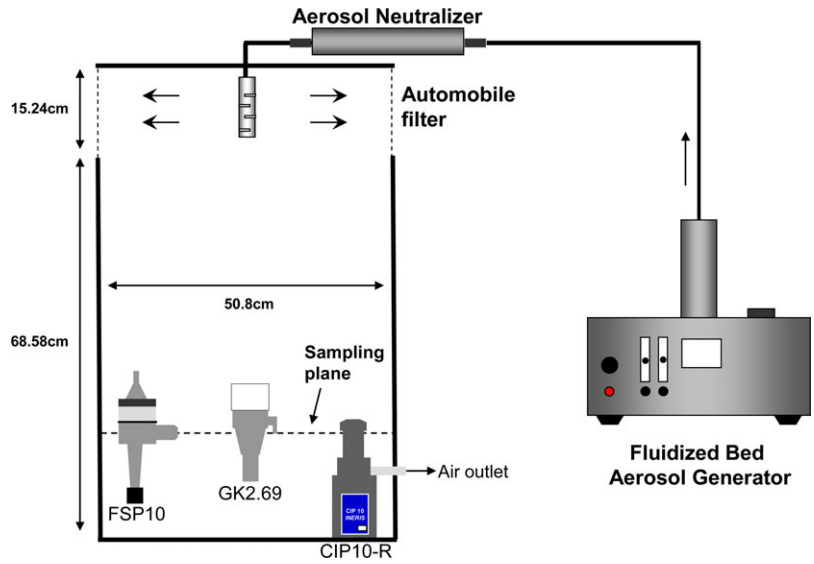

Fig. 1. Experimental setup for high flow rate samplers in a calm air chamber (B).

variation (CV) was calculated using average and standard deviation for each test.

Sampling efficiency test of high flow rate samplers

The sampling efficiency test for the high flow rate samplers was conducted in the calm air chamber A. The top plate of the chamber was modified to allow a ^{85}Kr aerosol neutralizer (Model 3054A; TSI Inc.) to be used in the dry column. In addition, instead of using the special nozzle utilized in chamber B, a baffle was hung 10 cm down from the top plate to disperse the aerosol. Eleven different sizes of monodisperse ammonium fluorescein aerosols were generated using a vibrating orifice aerosol generator (VOAG; Model 3450; TSI Inc.), fluorescein (Fisher Scientific), and ammonium hydroxide (Fisher Scientific) (Vanderpool and Rublow, 1988; Tsai and Shih, 1995). Particle size and generation rate were monitored with an APS. The aerodynamic diameters of each monodisperse aerosol were considered to be the geometric mean of each size distribution. A high flow rate respirable sampler and reference 25-mm open cowl sampler were placed horizontally inside of the chamber with collection media at the same sampling plane. The sampling media for high flow rate samplers were as described above and the reference samplers were loaded with 25-mm PVC filters (5 μm pore size; SKC Inc.). The air outlet of the CIP10-R was vented to the outside of the chamber in order to minimize disturbance of the calm air. Since the CIP10-R sampler is turned on and off by a magnet, it was modified in order to have the switch located outside of the chamber. The flow rate of CIP10-R was calibrated by using a CIP10 calibration bench (Arelco ARC).

The flow rates of the reference samplers were the same as the test sampler (i.e. 10 l min^{-1} for FSP10 and CIP10-R, and 4.2 l min^{-1} for GK2.69) and the inlet diameter for the reference sampler was calculated using equation from Baron and Willeke (2001). The calculated minimum inlet diameters of reference samplers with the same flow rates as the GK2.69 and FSP10 samplers are 1.52 and 2.15 cm, respectively. The empirical equation of Grinshpun *et al.* (1993) was used to calculate the aspiration efficiency. The calculated aspiration efficiencies for the reference samplers were ∼100% for all particle sizes tested in this study.

After sampling, the collection media were placed in the 5% ammonium hydroxide solution to extract the fluorescein and the fluorescent intensities of the extracted fluorescein were measured by using a luminescence spectrometer (LS50B; PerkinElmer, Waltham, MA, USA). Each combination of particle size and sampler was run three times.

Sampling efficiency comparison to the international standard respirable convention

The sampling efficiencies for the high flow rate samplers were compared to the International Standards Organization (ISO)/Comité Européen de Normalisation (CEN)/ACGIH respirable convention curve by calculating the bias. The estimated biases for each high flow rate sampler were calculated by

using equations (1–3) (Bartley *et al.*, 1994; Kenny and Bartley, 1995; Aitken *et al.*, 1999). Lognormal distribution was assumed and the calculation ranges of MMAD and GSD were 1–25 μm and 1.5–3.5, respectively. The bias of the sampler (Δ) is defined as

$$\Delta = \frac{\bar{C} - C_{\text{std}}}{C_{\text{std}}}, \tag{1}$$

$$\bar{C} = \int_0^\infty \bar{E}(D)A(D)dD, \tag{2}$$

where $\bar{C}$ is the mean measured concentration, $\bar{E}(D)$ is the mean sampling efficiency of the sampler, and $A(D)$ is the normalized size distribution.

$$C_{\text{std}} = \int_0^\infty \bar{F}(D)A(D)dD, \tag{3}$$

where C_{std} is the ideal concentration, $\bar{F}(D)$is the sampling efficiency of an ideal sampler, perfectly following the desired sampling convention.

Dust loading effect on the high flow rate cyclones

Although cyclones are known for low particle bounce and low re-entrainment, the accumulation of deposited particles inside the cyclone might significantly interfere with and alter the aerosol flow (Chen and Huang, 1999). In order to test the dust loading effect on the sampling efficiency for the high flow rate cyclones, sampling efficiency was determined using ammonium fluorescein monodisperse particles in calm air chamber A at the size of 4 and 5 μm before and after dust loading with AC fine test dust (Duke Scientific) in calm air chamber B. Three different mass concentrations, i.e. below the ACGIH respirable dust TLV (3 mg m^{-3}), around the TLV, and over the TLV (between two and three times of TLV), were used for this experiment and samplers were loaded for ~180 min in each test. Three repetitions of each test were conducted and the exact mass concentrations were determined by gravimetric analysis of the filter from chamber B. Since the CIP10-R is not a cyclone sampler and pressure drop across foam is much smaller than across a filter, this test was not conducted for this sampler.

Mass concentration comparison

In order to compare mass concentrations determined by the high flow rate samplers with those determined by the low flow rate cyclones in common use in the USA [10-mm nylon cyclone (Dorr-Oliver; Sensidyne, Clearwater, FL, USA) and HD cyclone (Model BGI-4; BGI Inc.)], one of each of all five samplers was placed in the calm air chamber B and challenged with three different aerosols, including aluminum oxide grades F800 and F1200 and AC fine dust. The specific sampler was randomly selected from several different units for this test. The mass concentrations were calculated from the gravimetric analysis and sampled air volume. Both the mass concentration ratio and net mass ratio were calculated. The mass concentration ratio was defined as the mass concentration from each high flow rate sampler divided by the mass concentration measured with either the nylon cyclone or the HD cyclone. The net mass ratio was defined as mass collected by each high flow rate sampler divided by the mass collected by the nylon cyclone or HD cyclone.

Statistical analysis

Data were analyzed to find differences between mean levels of sampling efficiency before and after dust loading for 180 min. Although the data are expected to be normally distributed, there were only three data points for each group and the standard deviations were estimated from the data, so *t*-tests were used for the comparisons. Pooled standard deviations were used to calculate the *t*-statistic as they were found to be unequal between groups. Differences in mean efficiency levels between before and after dust loading were considered significant at an α level of 0.05, and 95% confidence intervals were generated.

RESULTS

Uniformity test of the calm air chamber

For three different particles including aluminum oxide grades F800 and F1200 and kaolin, the average mass concentration, standard deviation, and CV of each test are shown in Table 3. The CVs ranged between 1.96 and 6.11% for nine different tests, which indicated that this calm air chamber B provided sufficiently uniform distribution for evaluation of the high flow rate samplers.

Sampling efficiency of high flow rate samplers

The sampling efficiency curves for CIP10-R, GK2.69, and FSP10 samplers from the present study and previous studies along with ISO/CEN/ACGIH respirable convention curve are shown in Fig. 2. The 50% cutoff size ($_{50}d_{\text{ae}}$) for the CIP10-R, GK2.69, and FSP10 samplers were 4.7, 4.1, and 4.8 μm, respectively. Figure 2 shows that the sampling efficiency of GK2.69 and FSP10 cyclones drops toward zero with increasing particle size more

Table 3. Calm air chamber uniformity test (mass, standard deviation, and CV)

Test aerosol	Test	Average mass concentration (mg m^{-3})	Standard deviation	CV (%)
Aluminum oxide (F1200)	1	142	5.72	4.04
	2	178	7.70	4.33
	3	117	3.28	2.81
Aluminum oxide (F800)	1	87	3.37	3.86
	2	91	5.53	6.11
	3	69	3.77	5.53
Kaolin	1	30	1.75	5.79
	2	56	1.11	1.96
	3	50	2.05	4.12

sharply than the ISO/CEN/ACGIH respirable convention curve does. The normal practice for comparing samplers to a size-selection standard is through the use of bias maps. Assuming workplace aerosols are unimodal with a lognormal distribution, they can be described by the MMAD and GSD. For any combination of these two values, a difference in collection efficiency can be calculated for the tested sampler and an ideal sampler that performs exactly in accordance with the size-selection standard. A plot of bias for all points in a space bounded by reasonable ranges of MMAD and GSD is called a bias map. The objective of sampler calibration is to find the flow rate that most minimizes the bias rather than to best meet the ${}_{50}d_{ae}$. The bias maps for each high flow rate sampler are shown in Fig. 3. The estimated biases were positive for all the size distributions implying that all the high flow samplers collected more dust than the ISO/CEN/ACGIH respirable convention.

Dust loading effect on the high flow rate cyclones

Table 4 shows the sampling efficiencies of the GK2.69 and FSP10 cyclones before and after 3 h dust loading and the mass concentration for each test. Test 2 at 4 μm and Test 1 at 5 μm for GK2.69 cyclone gave statistically significant different performances before and after 3 h dust loading. For the FSP10 cyclone, Test 1 at 4 μm and Test 2 at 5 μm showed statistically significant differences. These inconsistent differences were considered as a reflection of experimental random error (sampler and experiment variance) rather than an indication that the sampling efficiency changes after dust loading inside of the cyclone. Chen and Huang (1999) showed that the sampling efficiency decreased ~20% when the 10-mm Nylon (SKC), Multi-inlet, and Bigger Body (a cyclone constructed with a bigger body diameter than 10-mm nylon cyclone) cyclones were exposed to potassium sodium tartrate. In the same study, however, it was also observed that the change of the sampling efficiency depended on the nature of the challenge particles.

Mass concentration comparison

Table 5 shows mass concentration and net mass ratios of CIP10-R, GK2.69, and FSP10 samplers to the 10-mm nylon and HD cyclones, obtained by challenge from three different sizes of particles. The mass concentration ratios of FSP10 to 10-mm nylon cyclone and HD cyclone were the largest (1.85 and 1.54, respectively) with the aluminum oxide grade F800 test aerosol, followed by those of the CIP10-R and GK2.69. The mass concentration ratio decreased when the MMAD decreased for all the samplers. The difference of mass concentration ratio between the samplers also decreased when the MMAD decreased. The CIP10-R sampler underestimated the concentration up to 24% compared to the HD cyclone when the samplers were exposed to the AC fine dust.

According to the net mass ratio results, the CIP10-R collected between five and nine times more than 10-mm nylon cyclone and between three and six times more than HD cyclone. The GK2.69 cyclone collected approximately three and two times more than 10-mm nylon and HD cyclones, respectively. The FSP10 collected between 7 and 11 times more mass than 10-mm nylon cyclone and between 5 and 7 times more mass than HD cyclone with the challenged particles. The HD cyclone collected approximately 1.6 times more than 10-mm nylon cyclone.

DISCUSSION

Sampling efficiency test of high flow rate samplers

CIP10-R. The CIP10-R underestimated in the size range between 1 and 2.5 μm and overestimated particles >2.5 μm when compared with the ISO/CEN/ACGIH respirable convention curve. The sampling efficiency of the CIP10-R differed from that of cyclones in the size range between 1 and 2.5 μm because the CIP10-R sampler fails to capture some fraction of fine particles. The overall shape of the sampling efficiency curve was similar to that observed in previous studies (Courbon *et al.*, 1988; Görner, Wrovel, Micka, 2001). Those studies utilized different particles and methods for

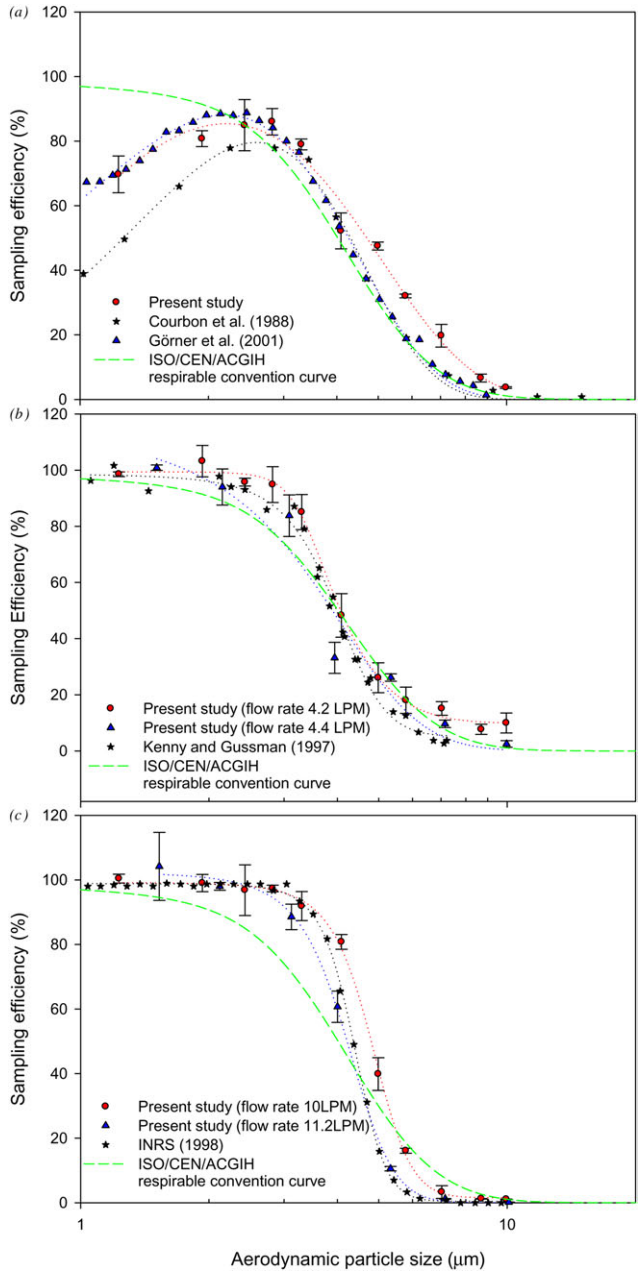

Fig. 2. Sampling efficiency for CIP10-R (a), GK2.69 (b), and FSP10 (c) samplers from present study, from previous studies for each sampler, and ISO/CEN/ACGIH respirable convention.

determination of sampling efficiency, i.e. Courbon *et al.* utilized coal dust and Aloxite 50 aerosols and with a Coulter Counter and Görner *et al.* utilized ground coal dust and an APS.

GK2.69. Kenny and Gussman (1997) investigated the sampling efficiency of the GK2.69 cyclone by using an APS with solid spherical glass microspheres up to 7 μm aerodynamic equivalent diameter. The sampling efficiency curves from both that study and this (Fig. 2b) were very similar for particle sizes <6 μm and the $_{50}d_{ae}$ are almost the same. However, it was noticeable that the sampling

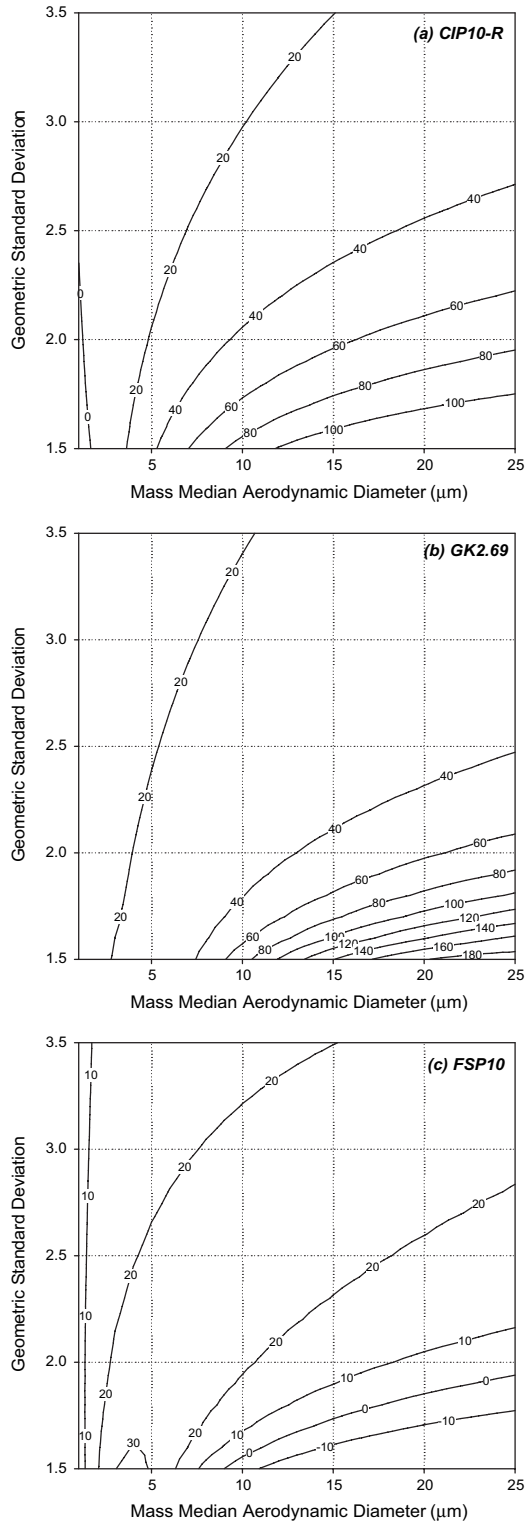

Fig. 3. Bias maps of CIP10-R (a), GK2.69 (b), and FSP10 (c) samplers.

efficiency at 10 μm from the present study was near 9.9% while that of the respirable convention is near 1%. This discrepancy created large bias for particles having large MMAD (Fig. 3b). The sampling efficiency at 7 μm from the Kenny and Gussman study was ∼3%. In addition, in the aerosol sampler testing exchange (ASTEX) study (Kenny 2000; Lidén 2000), six different laboratory tests of sampling efficiency of the GK2.69 were determined by APS and most were <5% sampling efficiency ∼10 μm. In order to rule out inter-sampler variation in the present study, the sampling efficiencies of three different GK2.69 samplers were determined using 10 μm ammonium fluorescein monodisperse particles; the sampling efficiencies were found to be 8.1, 5.5, and 8.6%. To rule out the possibility of contamination through the procedure and to check the particle size, the ∼10-μm ammonium fluorescein monodisperse particles were generated and collected on polycarbonate filters in both GK2.69 and reference samplers. Particles were observed by a field emission scanning electron microscope. The particle sizes of the ammonium fluorescein were between 8 and 9 μm (when the density was applied to calculate the aerodynamic particle diameter, the particle size was quite close to the APS measurement) and several agglomerated particles were observed on the cyclone filter that were not seen on the reference sampler filter. The reason for this agglomeration in the cyclone was not identified. The numbers of particles per microscopic field were counted in both samples by visual inspection and those were estimated to be commensurate with a 10% sampling efficiency for the GK2.69 cyclone. Another sampling efficiency curve for the GK2.69 cyclone was determined by the APS using grade F800 aluminum oxide and the result is shown in Fig. 4 along with that determined by ammonium fluorescein monodisperse particles. The sampling efficiency curve from the APS showed similar results to the Kenny and Gussman study at 10-μm particle size. However, the $_{50}d_{ae}$ determined by the APS was smaller than that determined by the ammonium fluorescein intensity method. The difference between the two methods might be attributed to the density correction for the APS (Wang and John, 1987) indicating that the individual particle density is smaller than the bulk density. In addition, the shapes of the particles are different (i.e. ammonium fluorescein particles are spheres and the aluminum oxide particles are irregular shapes) and the APS might thus underestimate the size of the aluminum oxide (Cheng *et al.*, 1990). This discrepancy could also be attributed to the coincidence effect of the

Table 4. Sampling efficiency at initial and 3 h dust loading for FSP10 and GK2.69

Sampler	Particle size[a] (μm)	Test	Sampling efficiency (%) at time 0 min	Loaded dust concentration (mg m^{-3})	Sampling efficiency (%) at time 180 min
GK2.69	4	1	27.2 ± 4.4	1.9	29.7 ± 4.3
		2[b]	36.7 ± 0.1	3.1	28.3 ± 0.6
		3	35.3 ± 6.0	7.9	38.1 ± 1.1
	5	1[b]	29.7 ± 1.9	1.9	25.1 ± 0.8
		2	28.7 ± 1.3	3.1	20.9 ± 6.1
		3	25.6 ± 3.9	7.9	24.9 ± 4.4
FSP10	4	1[b]	75.3 ± 1.0	2.3	67.6 ± 1.7
		2	70.8 ± 4.8	3.3	71.7 ± 5.2
		3	73.0 ± 8.2	8.4	76.4 ± 7.0
	5	1	27.2 ± 4.4	2.3	20.1 ± 0.9
		2[b]	45.0 ± 4.7	3.3	34.9 ± 3.3
		3	25.8 ± 2.5	8.4	24.9 ± 2.8

Sampling efficiency (%) are average and standard deviation of triplicate measures. The loaded dust concentration in Test 1 is below ACGIH respirable dust TLV (3 mg m^{-3}), in Test 2 is around TLV, and in Test 3 is two or three times of TLV.
[a]Particle size was measured with an APS.
[b]Statistically significant before and after dust loading at an α level of 0.05.

Table 5. Mass concentration ratio and net mass ratio of high flow rate samplers to nylon cyclone

	Dust type	Reference cyclone	CIP10-R	GK2.69	FSP10
Mass concentration ratio[a]	F800	10 mm nylon	1.52 ± 0.24	1.38 ± 0.08	1.85 ± 0.11
		HD	1.26 ± 0.09	1.15 ± 0.11	1.54 ± 0.13
	F1200	10 mm nylon	1.21 ± 0.11	1.28 ± 0.06	1.50 ± 0.07
		HD	0.91 ± 0.08	0.97 ± 0.02	1.14 ± 0.05
	AC fine dust	10 mm nylon	0.89 ± 0.10	1.12 ± 0.05	1.17 ± 0.06
		HD	0.76 ± 0.05	0.95 ± 0.01	0.99 ± 0.01
Net mass ratio[b]	F800	10 mm nylon	8.89 ± 1.41	3.41 ± 0.21	10.76 ± 0.60
		HD	5.77 ± 0.46	2.22 ± 0.17	7.04 ± 0.54
	F1200	10 mm nylon	7.10 ± 0.70	3.13 ± 0.14	8.51 ± 0.38
		HD	4.16 ± 0.45	1.84 ± 0.06	4.98 ± 0.25
	AC fine dust	10 mm nylon	5.24 ± 0.58	2.73 ± 0.12	6.85 ± 0.37
		HD	3.45 ± 0.26	1.80 ± 0.53	4.52 ± 0.09

The mass concentration and net mass ratios are average and standard deviation of triplicate measures.
[a]Mass concentration ratio is defined as the mass concentration of each sampler to mass concentration of the reference cyclones (10-mm nylon and HD cyclones).
[b]Net mass ratio is defined as the net mass of each sampler to net mass of the reference cyclones.

APS, underestimation of particles >5 μm of APS, and large particle loss in tubing (Pagels *et al.* 2005). Sampling efficiency of the GK2.69 cyclone was determined with a change in flow rate to 4.4 l min^{-1} and it is shown in Fig. 2b. The sampling efficiency at ~10 μm was found to be 2.5 ± 1.2% in triplicate tests.

FSP10. The FSP10 sampler overestimated the exposure in the size range between 1 and 5.5 μm compared to the ISO/CEN/ACGIH respirable convention curve. The $_{50}d_{ae}$ of the FSP10 from present study is greater than that observed by the Institut National de Recherche et de Sécurité (INRS, unpublished data) (4.35 μm, in Fig. 2a). This difference might arise from the different test methods and particles. A previous study from Health and Safety Laboratory (Cossey and Vaughan, 1987) using a prototype that was the basis for the FSP10 cyclone determined that

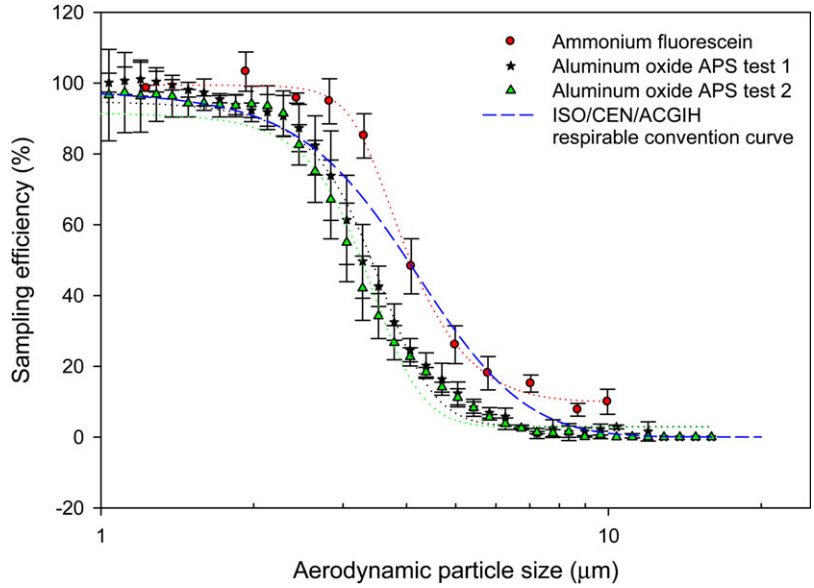

Fig. 4. Sampling efficiency curves for GK2.69 sampler determined by APS (Tests 1 and 2 challenged with aluminum oxide grade F500) and ammonium fluorescein monodisperse particles.

the sampling efficiency measured with the APS matched the British Medical Research Council convention ($_{50}d_{ae}$ = 5 μm) at a flow rate of 9.2 l min^{-1}. Cossey and Vaughan reported an equation (4) for the calculation of the $_{50}d_{ae}$.

$$d_{50}(\mu\text{m}) = \frac{68.7}{Q^{1.15}(1\ \text{min}^{-1})}. \quad (4)$$

The $_{50}d_{ae}$ at 10 l min^{-1} calculated using equation (4) is 4.86 μm, which is quite close to that found in the present study. When the flow rate is tuned to 11.2 l min^{-1} in order to better match the ISO/CEN/ACGIH respirable convention curve, the sampling efficiency curves are closer to the respirable convention curve than that at 10 l min^{-1} (Fig. 2c).

The sampling efficiency of the samplers may be different in moving air. Görner, Simon, Wrobel, (2010) investigated sampling efficiencies of six different inhalable samplers in a wind tunnel with velocity 1 ms^{-1} and in a calm air chamber. They found that the sampling efficiency was significantly lower in a wind tunnel than in a calm air chamber for all tested samplers. However, a calm air environment would be closer to realistic working environments (average air velocity <0.3 m s^{-1}) according to Baldwin and Maynard (1998).

Sampling efficiency comparison to the international standard

The estimated biases presented here for each high flow rate sampler are greater than those found in previous studies for the GK2.69 and CIP10-R samplers. Page (2003) reported size distributions of underground coal mine dust from 13 different locations by using personal cascade impactors. The MMAD size range of underground coal mine dust was 0.9–3.6 μm and the GSD was <2.37. An NIOSH publication (Coene, 1981) reported that the mean diameters of the dust particles at various silica flour mill operations were between 2.3 and 5.2 μm. A construction site field study (Shepherd *et al.*, 2009) reported MMAD of occupational dust from concrete-cutting hammer drills as 27.5 μm. Based on our laboratory results with the three samplers, a positive bias of ~25% for first two previous studies and 40% for the last study could be expected when sampling these occupational aerosols compared to the ISO/CEN/ACGIH respirable convention. Therefore, all three high flow rate samplers with current recommended flow rates might overestimate personal exposures. EN13205 (Workplace atmospheres: assessment of performance of instruments for measurement of airborne particle concentration) states that a sampler is in conformity with the relevant EN481 convention when the accuracy is ≤30% (the accuracy is not exactly same as bias) and FSP10 may be in conformity based on our bias results (bias is <30% at most particle sizes). However, estimated bias maps were created for the sampling efficiencies of the GK2.69 and FSP10 cyclones at alternative flow rates and those are shown in Fig. 5. Biases <20% are observed after adjustment of the sampler flow rates to their optimal values.

Mass concentration comparison

For most samplers, when the particle size decreased, the mass concentration ratio decreased, which could be attributed to the different $_{50}d_{ae}$ between samplers. According to the sampling efficiency curve of the FSP10 from this study, the FSP10 collected more dust between 4 and 5 μm dust than the other samplers. This was further investigated by comparing mass-weighted size distribution of generated aerosol obtained from the APS and mass concentration of reference samplers and calculating the respirable fraction mass concentration as follows. The mass-weighted size distributions of aluminum oxide grade F800 were measured by the APS and the mass concentrations from the reference samplers that maintained the same flow rates as the high flow rate samplers during measurement (<5% difference before and after measurement) were determined. The mean sampling efficiency ($\bar{E}$) for each size of ammonium fluorescein monodisperse particles was multiplied by the normalized mass-weighted size distribution ($(dm/d\log Dp)/\sum m$) from the APS (each size bin was divided by total mass) in order to calculate the respirable mass fraction for each sampler.

$$\mathrm{RMF}_{\mathrm{sampler}} = \sum_{D=0}^{D=\max} \frac{(dm/d\log D)}{\sum m} \times \bar{E}(D). \quad (5)$$

The calculated respirable mass concentration for each sampler was calculated by adjusting $\mathrm{RMF}_{\mathrm{sampler}}$ to the mass concentration of reference sampler. The average respirable mass concentration ratios (RMCRs) of the CIP10-R, GK2.69, and FSP10 to the 10-mm nylon cyclone in triplicate tests were 1.55 ± 0.08, 1.31 ± 0.11, and 1.59 ± 0.20, respectively, which are comparable to the ratios from Table 5 (e.g. 1.52, 1.38, and 1.85). The RMCR was also calculated by using the sampling efficiency determined by the APS (in Fig. 4) and the RMCR for the GK2.69 and 10-mm nylon cyclones determined by three different methods are presented in Fig. 6. The RMCR from gravitational analysis and sampling efficiency determined by VOAG are quite close, whereas that from the sampling efficiency determined by the APS was quite different.

The mass concentration ratios between GK2.69 and HD cyclones were close to unity based on our results (Table 5), indicating that the current flow rate of the GK2.69 would be appropriate for sampling of fine particles. Since the bias may become excessive as particle size increases above MMAD of 6 μm, a slightly higher flow rate may provide more accurate estimates. The net mass ratios between high flow rate samplers to the 10-mm nylon and HD cyclones were also dependent on the particle size distribution. The high flow rate samplers collected more dust than 10-mm nylon and HD cyclones, which indicates that they might provide more silica for subsequent analysis assuming similar silica content in the dust. The advantage of high flow rate samplers in collecting a larger sample may be diminished for direct on-filter measurement of silica because of the dispersion of the sample over a wider area of filter than

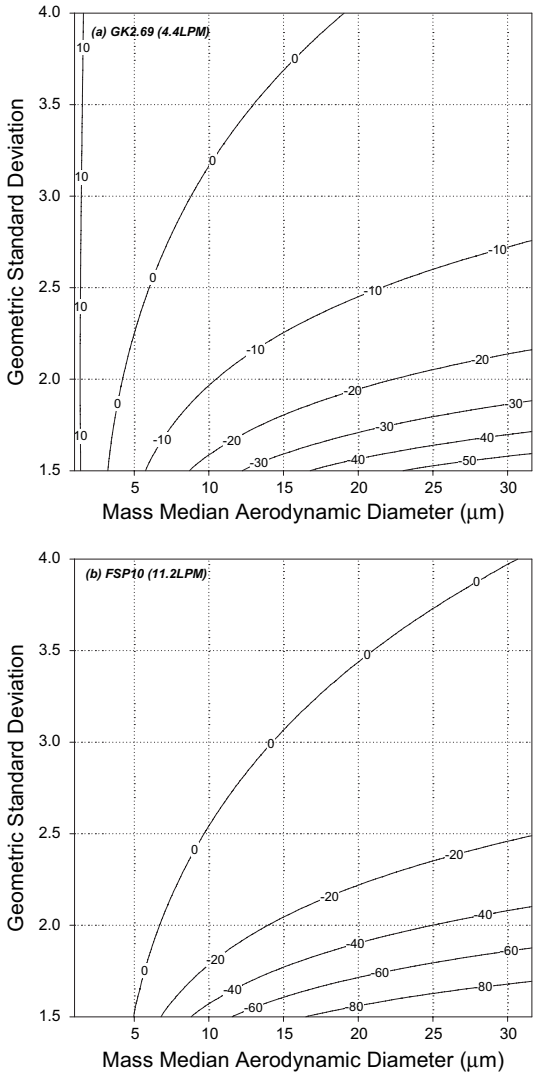

Fig. 5. Bias maps of GK2.69 (a) and FSP10 (b) samplers with adjusted flow rates.

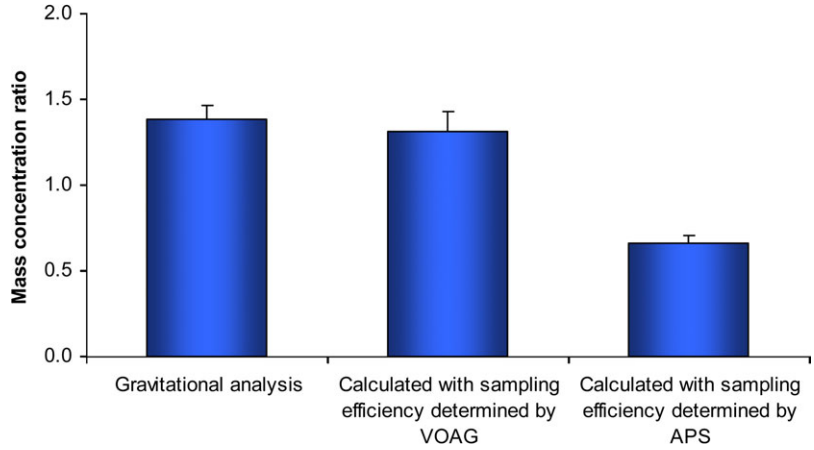

Fig. 6. RMCRs (GK2.69 cyclone to 10-mm nylon cyclone) comparison determined by three different methods.

would occur with a sampler using a 25-mm diameter filter.

CONCLUSIONS

The performances of high flow rate samplers were evaluated in this study. The sampling efficiency curves for CIP10-R, GK2.69, and FSP10 were determined with ammonium fluorescein monodisperse particles and compared with the ISO/CEN/ACGIH respirable convention. The high flow rate samplers overestimated exposure to respirable particles compared to the ISO/CEN/ACGIH respirable convention and could provide overestimated exposure data with the current flow rates recommended by manufacturers, especially, for example, with particles with MMAD >6 μm for the GK2.69 cyclone. However, both cyclones appeared to be able to provide relatively unbiased assessments of RCS if the flow rates are adjusted.

FUNDING

National Institute for Occupational Safety and Health project [Respirable silica measurements with high flow rate samplers (CAN# 0927ZGFR)].

Acknowledgements—Many thanks to Peter Stacey from Health and Safety Laboratory, UK, Carsten Möehlmann from Institute for Occupational Safety and Health, Germany, and Edmond Kauffer, Peter Görner, and Richard Wrobel from Institut National de Recherche et de Sécurité, France, for reviewing this work.

Disclaimer—The findings and conclusions in this report are those of the author(s) and do not necessarily represent the official position of the Centers for Disease Control and Prevention/the Agency for Toxic Substances and Disease Registry. Reference to the ACGIH TLV does not constitute endorsement by the US Federal Government.

REFERENCES

ACGIH. (2006) Threshold limit values (TLVs) and biological exposure indices (BEIs). Cincinnati, OH: American Conference of Governmental Industrial Hygienists.

Aitken RJ, Baldwin PEJ, Beaumont GC *et al*. (1999) Aerosol inhalability in low air movement environments. J Aerosol Sci; 30: 613–26.

Baldwin PEJ, Maynard AD. (1998) A survey of wind speeds in indoor workplaces. Ann Occup Hyg; 42: 303–13.

Baron P, Willeke K. (2001) Aerosol measurement. 2nd edn. Hoboken, NJ: Wiley Interscience; pp. 166–8.

Bartley DL, Chen CC, Song R *et al*. (1994) Respirable aerosol sampler performance testing. Am Ind Hyg Assoc J; 55: 1036–46.

Chen CC, Huang SH. (1999) Shift of aerosol penetration in respirable cyclone samplers. Am Ind Hyg Assoc J; 60: 720–9.

Cheng YS, Chen BT, Yeh HC. (1990) Behaviour of isometric nonspherical aerosol particles in the Aerodynamic Particle Sizer. J Aerosol Sci; 21: 701–10.

Coene RF. (1981) Silica flour: silicosis (crystalline silica). Cincinnati, OH: DHHS (NIOSH) Publication No. 81–137.

Cossey JR, Vaughan NP. (1987) A higher-flow rate cyclone for determination of respirable dust. Ann Occup Hyg; 31: 39–52.

Courbon P, Wrobel R, Fabries JF. (1988) A new individual respirable dust sampler: the CIP10. Ann Occup Hyg; 32: 129–43.

Dahmann D, Hartfiel G-D, Jackisch J. (2001) Intercomparison and performance of stationary aerosol samplers. Gefahrst Reinhalt Luft; 61: 201–6.

Eller PM, Feng HA, Song RS *et al*. (1999) Proficiency analytical testing (PAT) silica variability, 1990–1998. Am Ind Hyg Assoc J; 60: 533–9.

European Committee for Standardization (CEN). (2002) EN13205: Workplace atmospheres: assessment of performance of instruments for measurement of airborne particle concentrations. Brussels, Belgium.

Feather GA, Chen BT. (2003) Design and use of a settling chamber for sampler evaluation under calm-air conditions. Aerosol Sci Technol; 37: 261–71.

Gero A, Tomb T. (1988) Laboratory evaluation of the CIP10 personal dust sampler. Am Ind Hyg J; 49: 286–92.

Görner P, Simon X, Wrobel R *et al*. (2010) Laboratory study of selected personal inhalable aerosol samplers. Ann Occup Hyg; 54: 165–87.

Görner P, Wrovel R, Mička V *et al*. (2001) Study of fifteen respirable aerosol samplers used in occupation hygiene. Ann Occup Hyg; 45: 43–54.

Grinshpun S, Willeke K, Kalatoor S. (1993) A general equation for aerosol aspiration by thin-walled sampling probes in calm and moving air. Atmos Environ; 27: 1459–70.

Health and Safety Executive (HSE). (2002) Hazard assessment document, EH75/4. Respirable crystalline silica - phase 1: variability in fibrogenic potency and exposure-response relationships for silicosis. London: HSE Books.

International Agency for Research on Cancer (IARC). (1997) Silica, some silicates, coal dust and para-aramid fibrils. In Monograph on the evaluation of the carcinogenic risk of chemicals to humans. Vol. 68. Lyon, France: Working group on evaluation of carcinogenic risks to humans.

Kenny LC. (2000) Aerosol sampler testing exchange (ASTEX). J Aerosol Sci; 31 (Suppl. 1): S262–3.

Kenny LC, Bartley DL. (1995) The performance evaluation of aerosol samplers tested with monodisperse aerosols. J Aerosol Sci; 26: 109–26.

Kenny LC, Gussman RA. (1997) Characterization and modeling of a family of cyclone aerosol preseparators. J Aerosol Sci; 28: 677–88.

Lee T, Chisholm PW, Slaven EJ *et al*. (2009) Size distribution of 0.5 to 20 μm aerodynamic diameter lead-containing particles from aerosol samplers from aerosol sampler walls and filters. Aerosol Sci Technol; 43: 1042–50.

Lidén G. (2000) Penetration of the results from round 1 of ASTEX. J Aerosol Sci; 31 (Suppl. 1): S270–1.

Nij ET, Heederik D. (2005) Risk assessment of silicosis and lung cancer among construction workers exposed to respirable quartz. Scand J Work Environ Health; 31 (Suppl. 2): 49–56.

NIOSH. (2002) NIOSH hazard review: health effects of occupational exposure to respirable crystalline silica. Publication No. 2002-129; Cincinnati, OH: National Institute for Occupational Safety and Health.

NIOSH. (2003a). QUARTZ in coal mine dust, by IR (redeposition). In Paul Schlecht, Rosa Key-Schwartz, editors. NIOSH manual of analytical methods. 4th edn. Cincinnati, OH: National Institute for Occupational Safety and Health pp. 1–70.

NIOSH. (2003b). Silica, crystalline, by XRD (filter redeposition). In Rosa Key-Schwartz, Dawn Ramsey, Paul Schlecht, editors. NIOSH manual of analytical methods. 4th edn. Cincinnati, OH: National Institute for Occupational Safety and Health pp. 1–9.

Occupational Safety and Health Administration (OSHA). (2004) 1218-AB70-2040. Occupational exposure to crystalline silica. Available at http://www.osha.gov/pls/oshaweb/owadisp.show_document?p_table=UNIFIED_AGENDA&p_id=4506, URL. Accessed 25 June 2010.

Page SJ. (2003) Comparison of coal mine dust size distributions and calibration standards for crystalline silica analysis. Am Ind Hyg Assoc J; 64: 30–9.

Pagels J, Gudmundsson A, Gustavsson E *et al*. (2005) Evaluation of aerodynamic particle sizer and electrical low-pressure impactor for unimodal and bimodal mass-weighted size distributions. Aerosol Sci Technol; 39: 871–87.

Shepherd S, Woskie SR, Holcroft C *et al*. (2009) Reducing silica and dust exposures in construction during use of powered concrete-cutting hand tools: efficacy of local exhaust ventilation on hammer drills. J Occup Environ Hyg; 6: 42–51.

Soderholm SC. (1989) Proposed international conventions for particle size-selective sampling. Ann Occup Hyg; 33: 301–20.

Steenland K, Sanderson W. (2001) Lung cancer among industrial sand workers exposed to crystalline silica. Am J Epidemiol; 153: 695–703.

Tsai CJ, Shih TS. (1995) Particle collection efficiency of two personal respirable dust samplers. Am Ind Hyg Assoc J; 56: 911–8.

Ulm K, Gerein P, Eigenthaler J *et al*. (2004) Silica, silicosis and lung-cancer: results from a cohort study in the stone and quarry industry. Int Arch Occup Environ Health; 77: 313–8.

Vanderpool RW, Rublow KL. (1988) Generation of large, solid, monodisperse calibration aerosols. Aerosol Sci Technol; 9: 65–9.

Wang HC, John W. (1987) Particle density correction for the Aerodynamic Particle Sizer. Aerosol Sci Technol; 6: 191–8.